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IRON ORES - DETERMINATION OF VANADIUM BY ATOMIC ABSORPTION
STATISTICAL EVALUATION OF INTERNATIONAL TEST DATA - STAGE A - 1982

R. Sutarno and W.S. Bowman
Physical Chemistry Section
Extractive Metallurgy Laboratory

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IRON ORES - DETERMINATION OF VANADIUM BY ATOMIC ABSORPTION STATISTICAL EVALUATION OF INTERNATIONAL TEST DATA - STAGE A - 1982

by

R. Sutarno* and W.S. Bowman**

SUMMARY

The analytical data collected from the international test conducted by ISO/TC 102/SC 2/WG 18 A, on the determination of vanadium in iron ores by atomic absorption method during the period of 1980-1982 have been statistically evaluated.

Two variations of this method, (1) with extraction using 1-pentanol, and (2) without extraction, were investigated. It was found that the preliminary estimates of the precision can be expressed by the following linear equations.

With extraction	Without extraction
$r = 0.1224\ X + 0.0002$	$r = 0.0337\ X + 0.0038$
$P = 0.1766\ X + 0.0008$	$P = 0.1409\ X + 0.0038$
$\sigma_r = 0.0434\ X + 0.0001$	$\sigma_r = 0.0119\ X + 0.0013$
$\sigma_L = 0.0540\ X + 0.0003$	$\sigma_L = 0.0492\ X + 0.0011$
for $0.0004 \leq X \leq 0.055$	$0.061 \leq X \leq 0.543$

where r is the repeatability, P is the permissible tolerance, σ_r is the within-laboratories standard deviation and σ_L is the between-laboratories standard deviation, and X is the vanadium content of the sample, all in per cent. It was also found that improvement of the analytical method is necessary before it can be adopted as an international standard. A third international test is being conducted using the improved analytical procedure.

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*Research Scientist and **Technologist, Physical Chemistry Section, Extraction Metallurgy Laboratory, Mineral Sciences Laboratories, CANMET, Energy, Mines and Resources Canada, Ottawa, Ontario K1A 0G1.

MINERAIS EN FER. DÉTERMINATION DU VANADIUM PAR ABSORPTION ATOMIQUE ÉVALUATION STATISTIQUE PAR LE TEST INTERNATIONAL DE DONNÉES; ÉTAPE A-1982

par

R. Sutarno* et W.S. Bowman**

SOMMAIRE

On a évalué statistiquement des analyses rassemblées suite au test international dirigé par ISO/TC 102/SC 2/WG 18A, sur la détermination du vanadium dans les minerais en fer par la méthode d'absorption atomique qui a eu lieu pendant la période 1980-1982.

On a fait des recherches avec deux variétés de cette méthode: (1) avec l'extraction en utilisant 1-pentanol et (2) sans extraction. On a trouvé que l'évaluation préliminaire de cette précision peut être exprimée par les équations linéaires suivantes:

Avec extraction	Sans extraction
$r = 0,1224X + 0,0002$	$r = 0,0337X + 0,0038$
$P = 0,1766X + 0,0008$	$P = 0,1409X + 0,0038$
$\sigma_r = 0,0434X + 0,0001$	$\sigma_r = 0,0119X + 0,0013$
$\sigma_L = 0,0540X + 0,0003$	$\sigma_L = 0,0492X + 0,0011$
for $0,0004 \leq X \leq 0,055$	$0,061 \leq X \leq 0,543$

où r est la répétabilité, P est la tolérance permise, σ_r est l'écart-type à l'intérieur des laboratoires et σ_L est l'écart-type entre laboratoires, et X est la teneur de l'échantillon en vanadium; tous en pourcentage. On a aussi trouvé que l'amélioration de la méthode analytique était nécessaire avant qu'elle puisse être adoptée comme standard internationale. On est en train de diriger un troisième test international en utilisant la procédure analytique améliorée.

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*Chercheur scientifique et **Technologue, Section de chimie physique, Laboratoire de métallurgie extractive, Laboratoires des sciences minérales, CANMET, Energie, Mines et Ressources Canada, Ottawa, Ontario K1A 0G1.

INTRODUCTION

It was decided at the 10th meeting of ISO/TC 102/SC 2 that the development of a standard method for the determination of vanadium by atomic absorption be continued at stage A and a second international test be conducted using essentially the same procedure modified in light of the comments received at the meeting. The mandate of working group WG 18 A was tobe extended to carry out this development[1,2]. The procedure used for this international test is described in document ISO/TC 102/SC 2 N 700 E[3]. This international test was conducted during the period 1980-1982 with 16 laboratories participating. Eight test samples with vanadium content ranging from 0.004 to 0.5% were used and two variations in analytical procedure were tested. This report describes the full account of statistical evaluation of the results of this international test.

PRINCIPLE OF THE ANALYTICAL METHOD

Two variations of the method (referred to as Method 1 and Method 2) were tested. The principles are as follows:

Methods 1 and 2 - Decomposition of the test portion by digestion in glass with hydrochloric acid, transfer to a PTFE beaker, addition of hydrofluoric and nitric acids and evaporation to dryness. Addition of hydrochloric and perchloric acids and evaporation to dryness. Dissolution of the salts in perchloric and hydrochloric acids (Method 1), or in hydrochloric acid (Method 2), and filtration. Ignition and fusion of the residue with sodium carbonate and dissolution of the cooled melt in the test solution.

Method 1 - Oxidation with potassium permanganate solution and addition of phosphoric acid and sodium tungstate solutions. Extraction of the vanadium complex with 1-pentanol, treatment of the solvent phase with sodium hydroxide solution to strip the vanadium into water, and acidification of the aqueous phase with hydrochloric acid.

Methods 1 and 2 - Addition of aluminum solution to the test solution and dilution to volume. Aspiration into an acetylene-dinitrogen oxide flame in

an atomic absorption spectrometer and measurement of the absorbance at a wavelength of approximately 318.5 nm.

STATISTICAL EVALUATION

The analytical data were statistically evaluated by using the procedure described in document 2 N 532 E[4]. The factor 2.77 for conversion of standard deviations to their respective precision has been replaced by 2.83. This procedure is schematically illustrated in Figure 1. The raw data are plotted in Figure 2. The boxed points in this figure signify that these data are suspected outliers and were not used to estimate the grand means and precisions. The estimated precisions are plotted against their corresponding grand means in Figure 3. Linear regression equations of these lines were also computed and presented in Table 1. Table 2 lists the outlying laboratories and the ratios between the between-laboratories to the within-laboratory standard deviation. Table 3 shows the comparison between the grand means and the standard deviations obtained by the atomic absorption methods and those obtained by the spectrophotometric method.

DISCUSSION

From the results of this evaluation, the following are observed:

(1) Precision:

Table 1 shows that the within-laboratory precision is reasonable for the level of vanadium in the samples. The σ_L/σ_r ratios for method 2, however, are higher than normally acceptable for ISO standards (Table 2). For method 1, although the σ_L/σ_r ratios are acceptable, there is a laboratory (JP 1) that consistently had difficulty in maintaining accuracy (Dixon's test for outliers). This presumably indicates that further standardization of the method may be necessary. Table 3 shows only slight improvement in the precision from that obtained from the first international test using the procedure described in 2 N 556 E[3].

(2) Accuracy:

Table 3 shows that method 1 seems to produce higher results than those obtained by either the spectrophotometric method or by the first

international test. On the other hand method 2 seems to produce lower results.

ABBREVIATIONS USED IN THIS REPORT

σ_r = SIGMA-R - within-laboratory standard deviation

σ_L = SIGMA-L - between-laboratory standard devaiation

r* = R-repeatability

$= 2.83\ \sigma_r$

P** = permissible tolerance between-laboratories

$= 2.83\sqrt{\sigma_L^2 + \sigma_r^2/2}$

COUNTRY ABBREVIATIONS

AU = Australia

FR = France

IT = Italy

JP = Japan

NL = Netherlands

SW = Sweden

USA = United States of America

* Repeatability, r, is the value below which the absolute difference between two independent single test (analysis) results produced by a laboratory on identical material using the same equipment within a short interval of time, using the standardized test method, may be expected to be with a 95% probability.

** Permissible tolerance between laboratories, P, is the value below which the absolute difference between two means of duplicate test results on identical material obtained by operators of different laboratories using the standardized test method, may be expected to lie with a 95% probability.

BIBLIOGRAPHY

1. "Report of the tenth meeting of ISO/TC 102/SC - Iron Ores/Chemical Analysis, Pretoria, October 1980"; ISO/TC 102/SC 2/N 639 E; November 1980.

2. "IWG 18 - Determination of vanadium by atomic absorption method - Report of investigations to develop a minimum insoluble residue - Procedure for dissolution of a 2 g test portion"; ISO/TC 102/SC 2 (AU-21) N 627 E; September 1980.

3. "Determination of vanadium in iron ores and slags by atomic absorption spectrometry", ISO/TC 102/SC 2 (Netherland - 11) N 556 E; January 10, 1979.

4. "Procedure for statistical evaluation of analytical data resulting from international tests", ISO/TC 102/SC 2 (WG 7-21) N 532 E; October 1978.

5. "Report of Informal Working Group 18 on the determination of vanadium in iron ores by atomic absorption spectrophotometry (102/2 N 556 E)"; ISO/TC 102/SC 2 (IWG 18-6) N 611 E, September 1980.

6. "Iron Ores - Determination of vanadium content - BPHA Spectrophotometric method"; ISO/TC 102/SC 2 (WG 19B-5) N 698 E; September, 1982.

7. "Iron ores - Determination of vanadium content - Flame atomic absorption methods (Third working draft)"; ISO/TC 102/SC 2 (WG 18A-8) N 700 E; September 1982.

8. "Iron ores - Determination of vanadium by atomic absorption - Statistical analysis of international test data"; R. Sutarno and W.S. Bowman, MRP/MSL 81-102 (IR); July 1981.

9. "Iron ores - Determination of vanadium by spectrophotometric method - Statistical analysis of international test data - 1982"; R. Sutarno and W.S. Bowman, MRP/MSL 82-123 (IR); November 1982.

Table 1.1

Iron Ores - Vanadium by AAS method 1 (with extraction)
ISO/TC 102/SC 2/WG 18A-9

Summary of Precisions for all Samples

Sample	Mean(X)	Repeatability	Permissible tolerance	Sigma-R	Sigma-L
81-5	0.0550	0.0077	0.0103	0.00273	0.00306
79-14	0.0327	0.0029	0.0072	0.00101	0.00243
79-16	0.0052	0.0012	0.0019	0.00041	0.00060
79-17	0.0040	0.0010	0.0012	0.00035	0.00033

The regression equations are:		Corr. Coeff.
R	= 0.1224 X + 0.0002	0.9545
P	= 0.1766 X + 0.0008	0.9952
SIGMA-R	= 0.0434 X + 0.0001	0.9551
SIGMA-L	= 0.0540 X + 0.0003	0.9801

Table 1.2

Iron Ores - Vanadium by AAS method 2 (without extraction)
ISO/TC 102/SC 2/WG 18A-9

Summary of Precisions for all Samples

Sample	Mean(X)	Repeatability	Permissible tolerance	Sigma-R	Sigma-L
76-17	0.5427	0.0[illegible]28	0.0753	0.00805	0.02601
81-3	0.4020	0.0163	0.0673	0.00575	0.02343
81-4	0.1178	0.0077	0.0205	0.00273	0.00698
81-5	0.0611	0.0061	0.0103	0.00217	0.00328

The regression equations are:

		Corr. Coeff.
R	= 0.0337 X + 0.0038	0.9954
P	= 0.1409 X + 0.0038	0.9881
SIGMA-R	= 0.0119 X + 0.0013	0.9953
SIGMA-L	= 0.0492 X + 0.0011	0.9867

Table 2 - List of Outliers and Ratio of the between-laboratories standard deviation, σ_L, to the within-laboratories standard deviation, σ_r

Sample	Mean %	K_0	N_0	K	N	Outliers			σ_L/σ_r
						Cochran (precision)	Dixon (accuracy)	Result Individual	
						Method 1 (with extraction)			
81-5	0.055	16	64	14	56	FR 2*	JP 1	-	1.1
79-14	0.033	16	64	12	48	FR 1** SW 3**	FR 2 JP 1	- -	2.4
79-16	0.005	16	64	15	60	-	JP 1	-	1.5
79-17	0.004	16	63	13	51	SW 2 USA 2	JP 1	-	0.9
						Method 2 (without extraction)			
76-17	0.543	17	68	17	68	-	-	-	3.2
81-3	0.402	17	68	14	56	FR 3 IT 1 USA 2	-	-	4.1
81-4	0.118	17	68	15	60	AU 3 FR 3	-	-	2.6
81-5	0.061	17	68	17	68	-	-	-	1.5

K_0 - original numbers of sets of laboratory results
K - number of sets of laboratory results for ANOVA
N_0 - original number of results
N - number results used for ANOVA

* FR 2 for sample 81-5 were suspected outliers due to Cochran test for within-bottle variance

**FR 1 and SW 3 for sample 79-14 were rejected from two-way ANOVA due to zero within-bottle variance

Table 3. Comparison between the means and precision obtained from the International Test Data on the Determination of Vanadium by Spectrophotometric Method and Those Obtained by AAS Method

Sample	Mean by Spec. Method & 95% Confidence Interval			Mean by AAS Method & 95% Confidence Interval			σ_r		σ_L	
	Low	X	High	Low	X	High	Spec.	AAS	Spec.	AAS
				Method 1 (AAS With Extraction)						
81-5	0.05691	0.06001	0.06311	0.05264	0.05504*	0.05745	0.00117	0.00273	0.00424	0.00306
79-14	0.03197	0.03446	0.03696	0.03078	0.03265* (0.036)	0.03452	0.00062	0.00101 (0.0015)	0.00319	0.00243 (0.0013)
79-16	0.00508	0.00594	0.00679	0.00487	0.00523 (0.0058)	0.00558	0.00095	0.00041 (0.0005)	0.00124	0.00060 (0.0007)
79-17	0.00366	0.00469	0.00572	0.00377	0.00400 (0.0044)	0.00423	0.00032	0.00035 (0.0005)	0.00120	0.00033 (0.0003)
				Method 2 (AAS Without Extraction)						
76-3	0.27869	0.29548	0.31228	———	———	———	0.00548	———	0.02485	———
76-17	0.50506	0.51882	0.53259	0.52918	0.54271 (0.507)	0.55624	0.00886	0.00805 (0.031)	0.01735	0.02601 (0.042)
81-3	0.35855	0.37513	0.39171	0.38832	0.40195	0.41557	0.00802	0.00575	0.02283	0.02343
81-4	0.10983	0.11457	0.11930	0.11388	0.11782	0.12176	0.00335	0.00273	0.00685	0.00698
81-5	0.05691	0.06001	0.06311	0.05929	0.06107	0.06284	0.00117	0.00217	0.00424	0.00328

*Between-bottles variation is statistically significant
Bracketed values were obtained from the first international test using the procedure described in document 2 N 556 E.

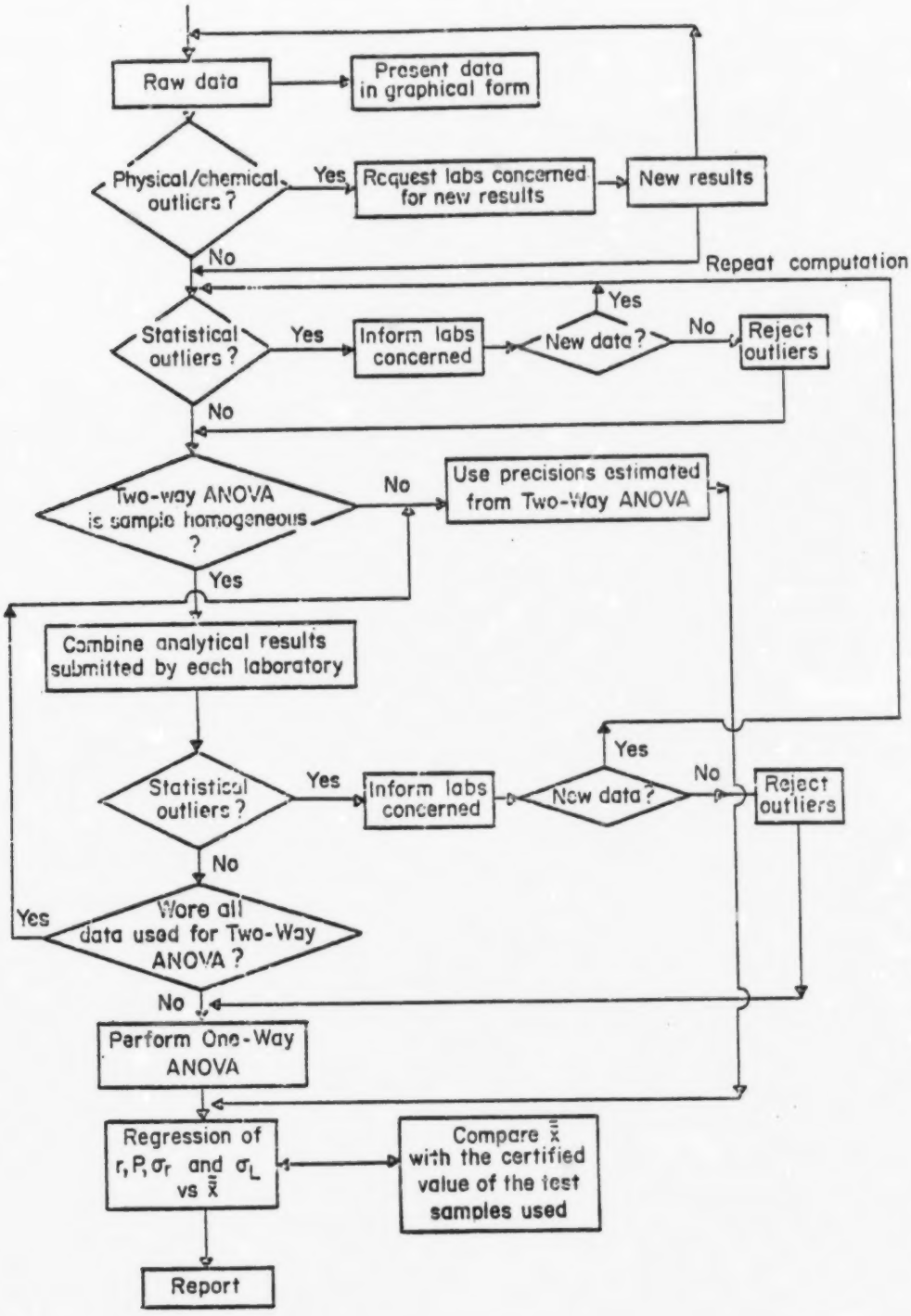

Figure 1. Flow Chart of the Procedure for Statistical Evaluation of Analytical Data Resulting from International Tests.

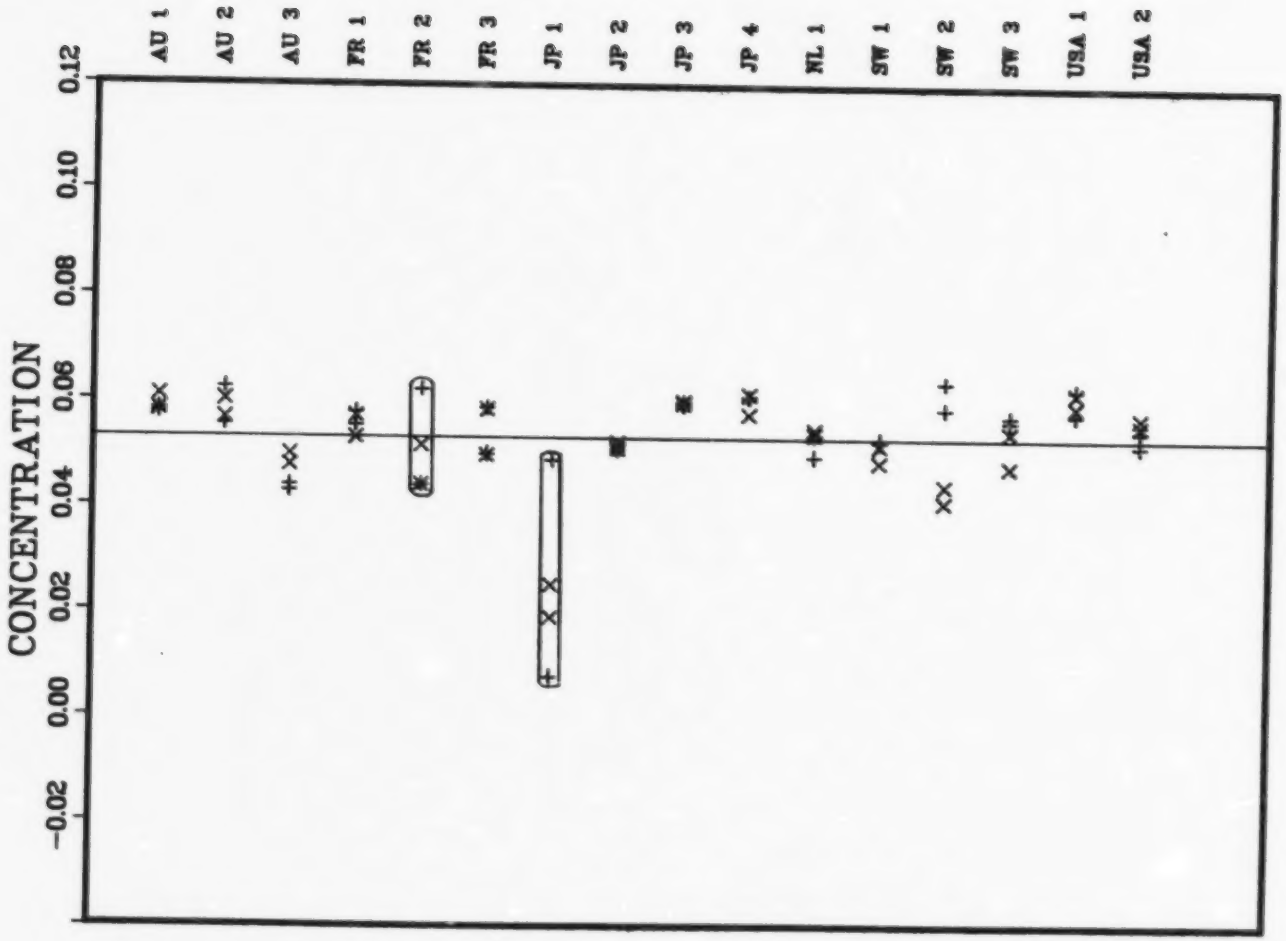

Figure 2.1.1 IRON ORES – VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9 SAMPLE 81-5

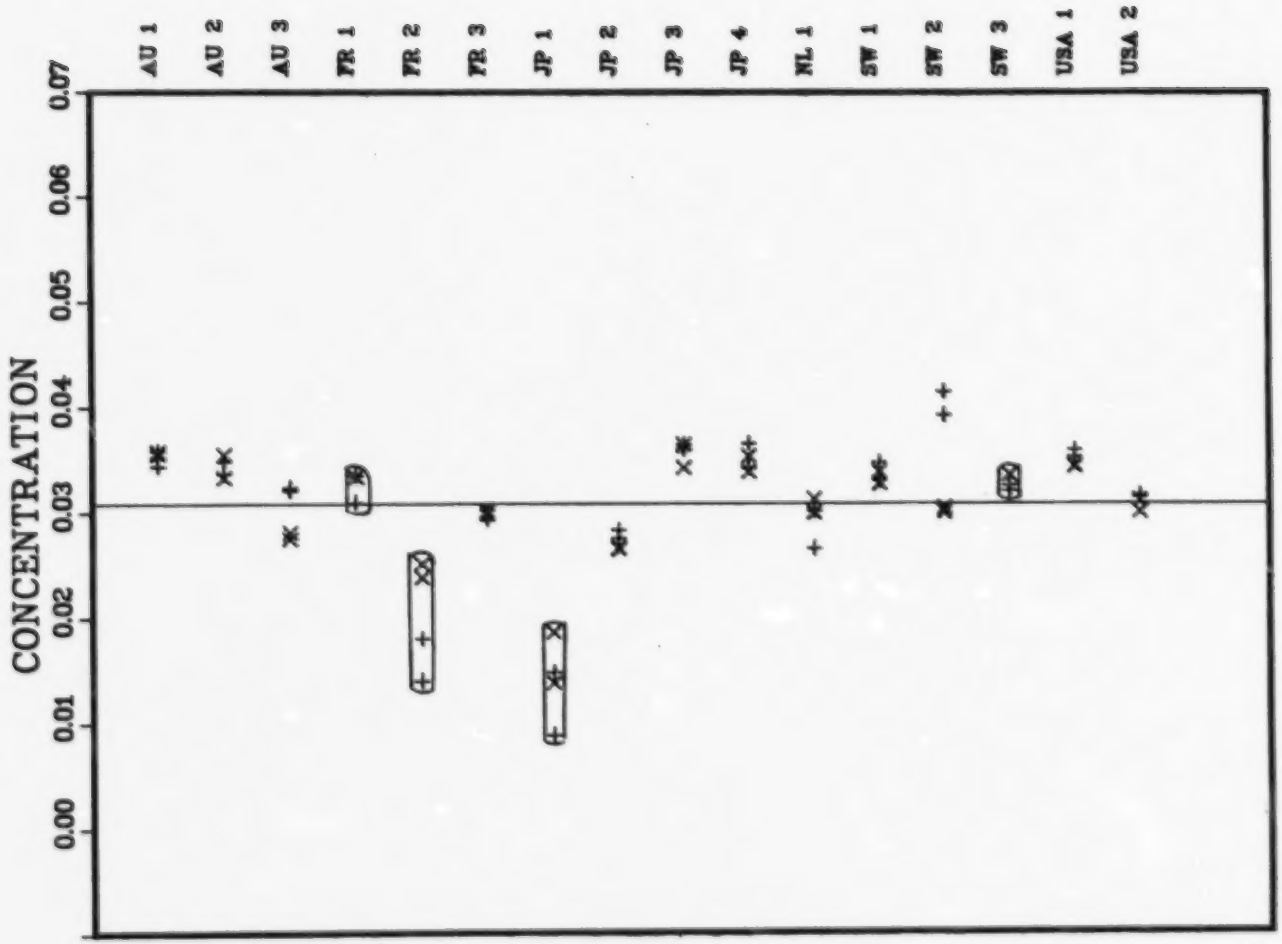

Figure 2.1.2 **IRON ORES – VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9 SAMPLE 79-14**

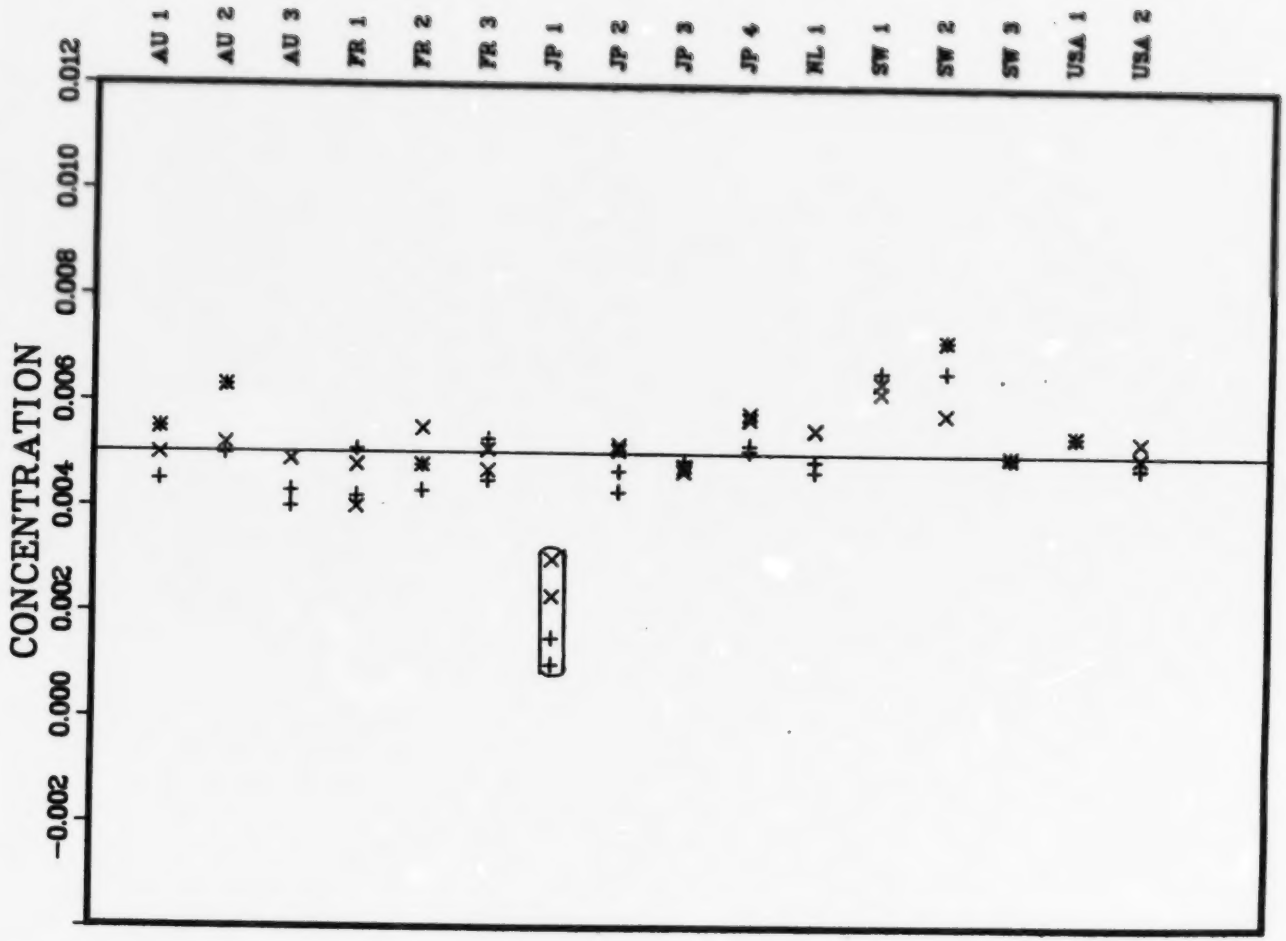

Figure 2.1.3 **IRON ORES – VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A–8 SAMPLE 79–16**

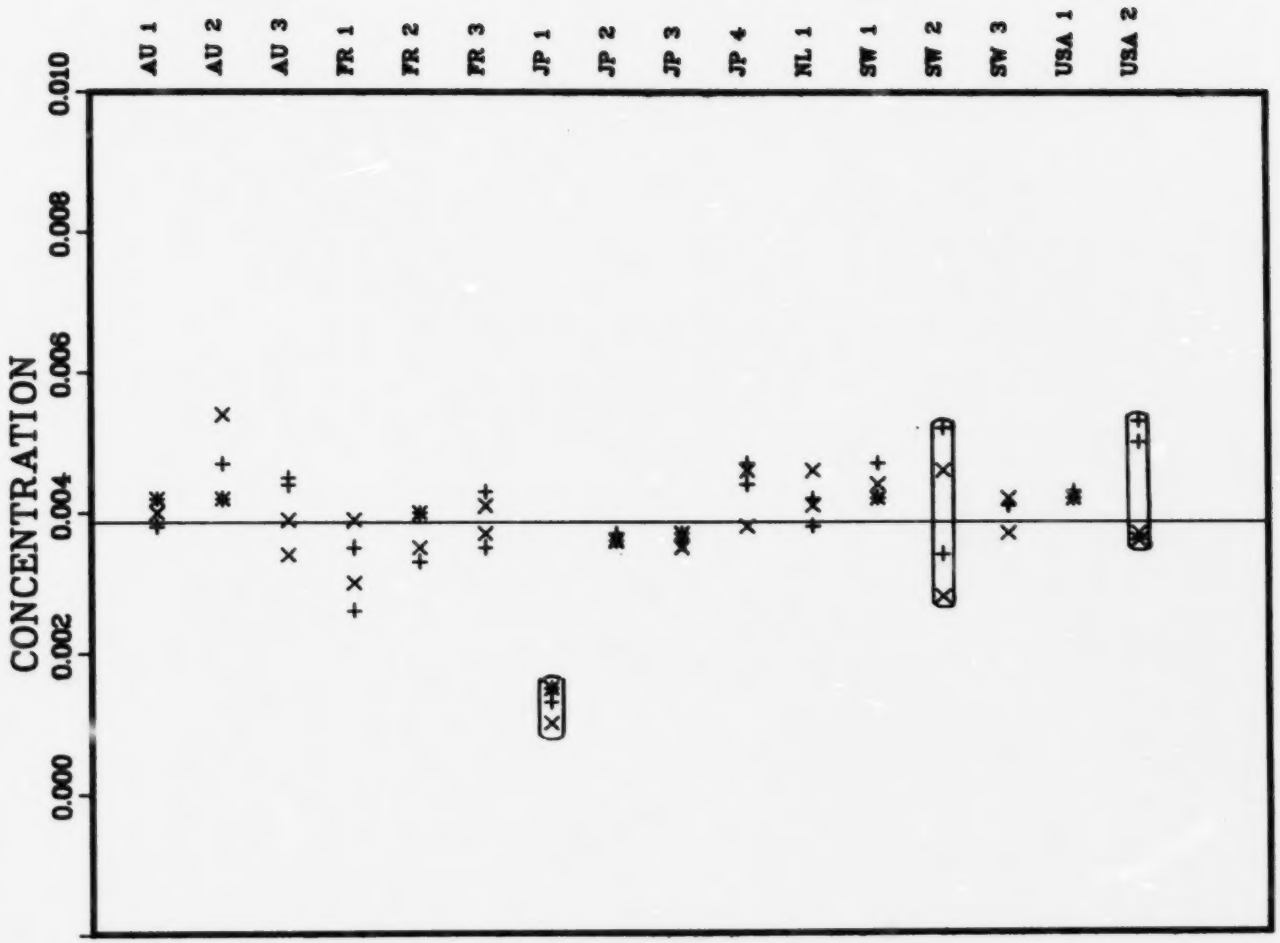

Figure 2.1.4 **IRON ORES – VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A–9 SAMPLE 79–17**

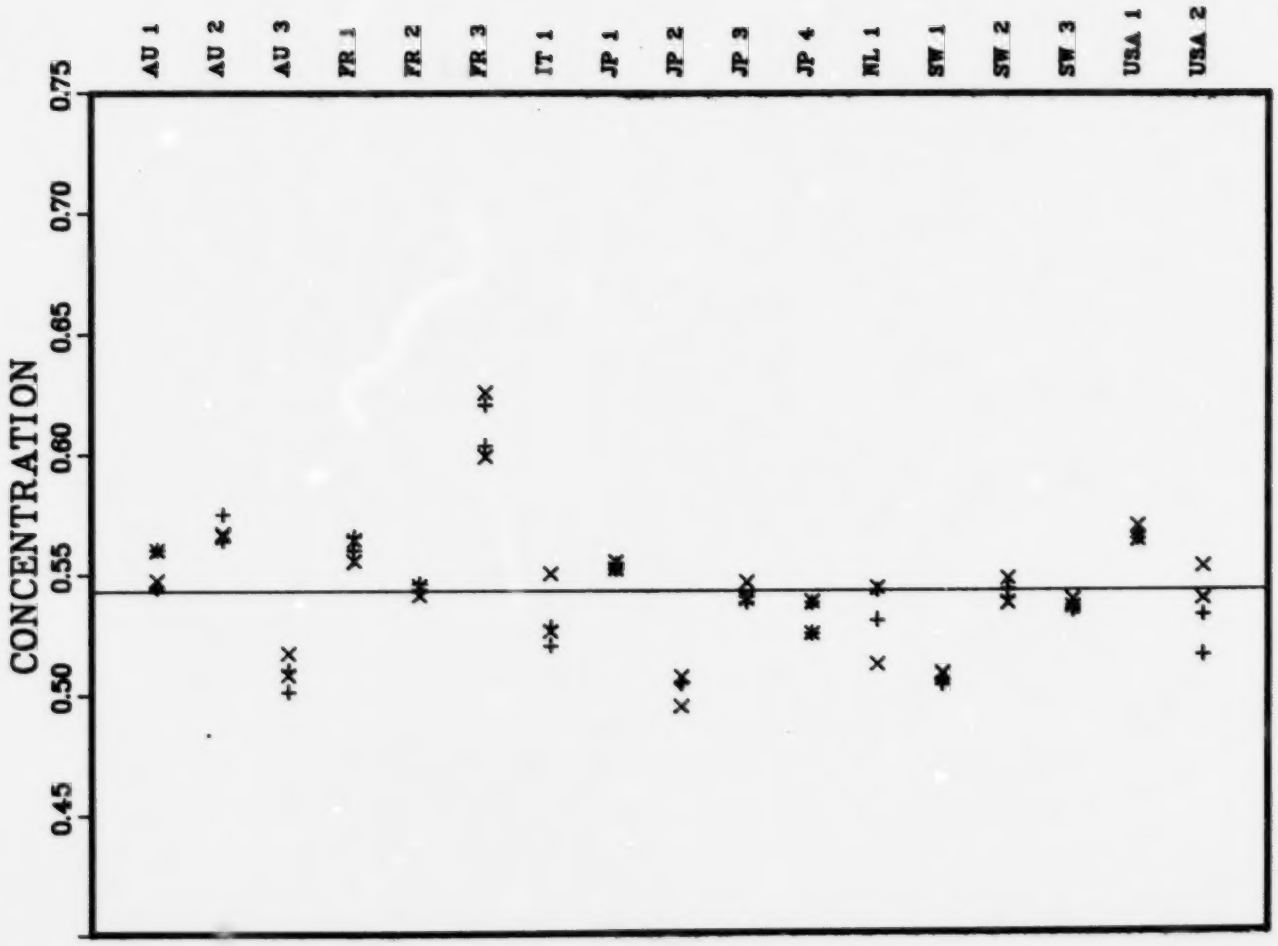

Figure 2.2.1 IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 76-17

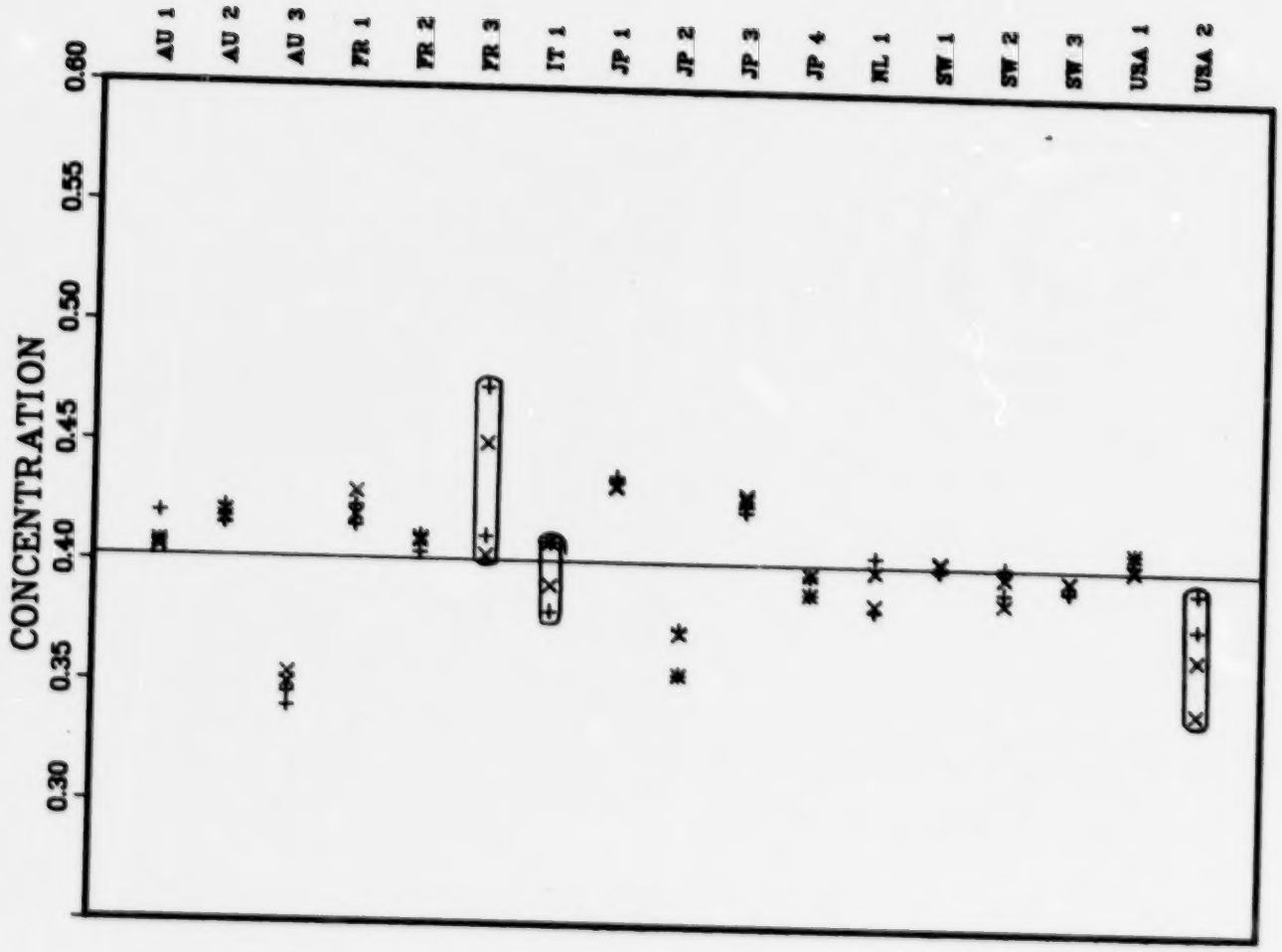

Figure 2.2.2 IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9 SAMPLE 81-3

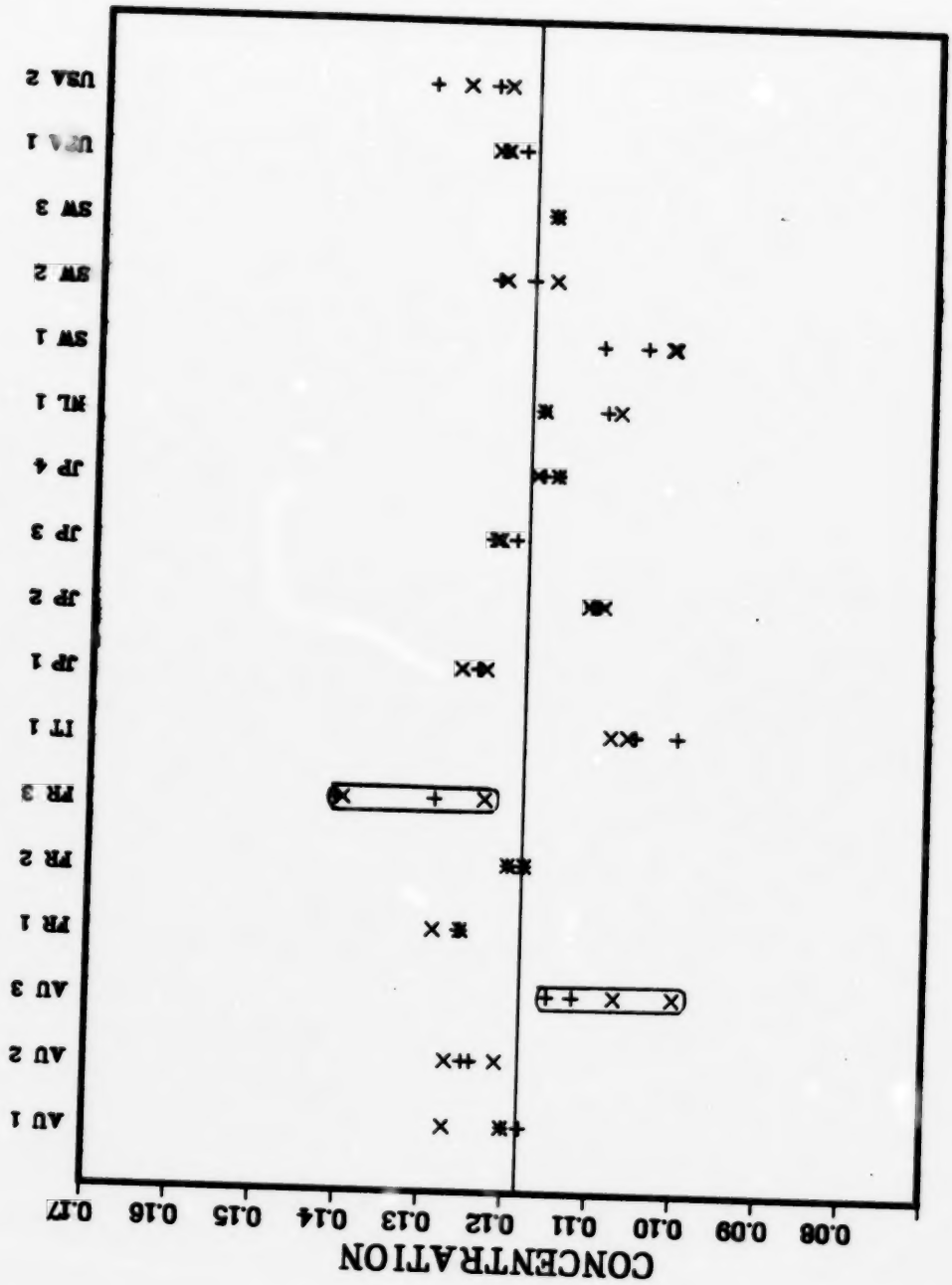

Figure 2.2.3 IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-4

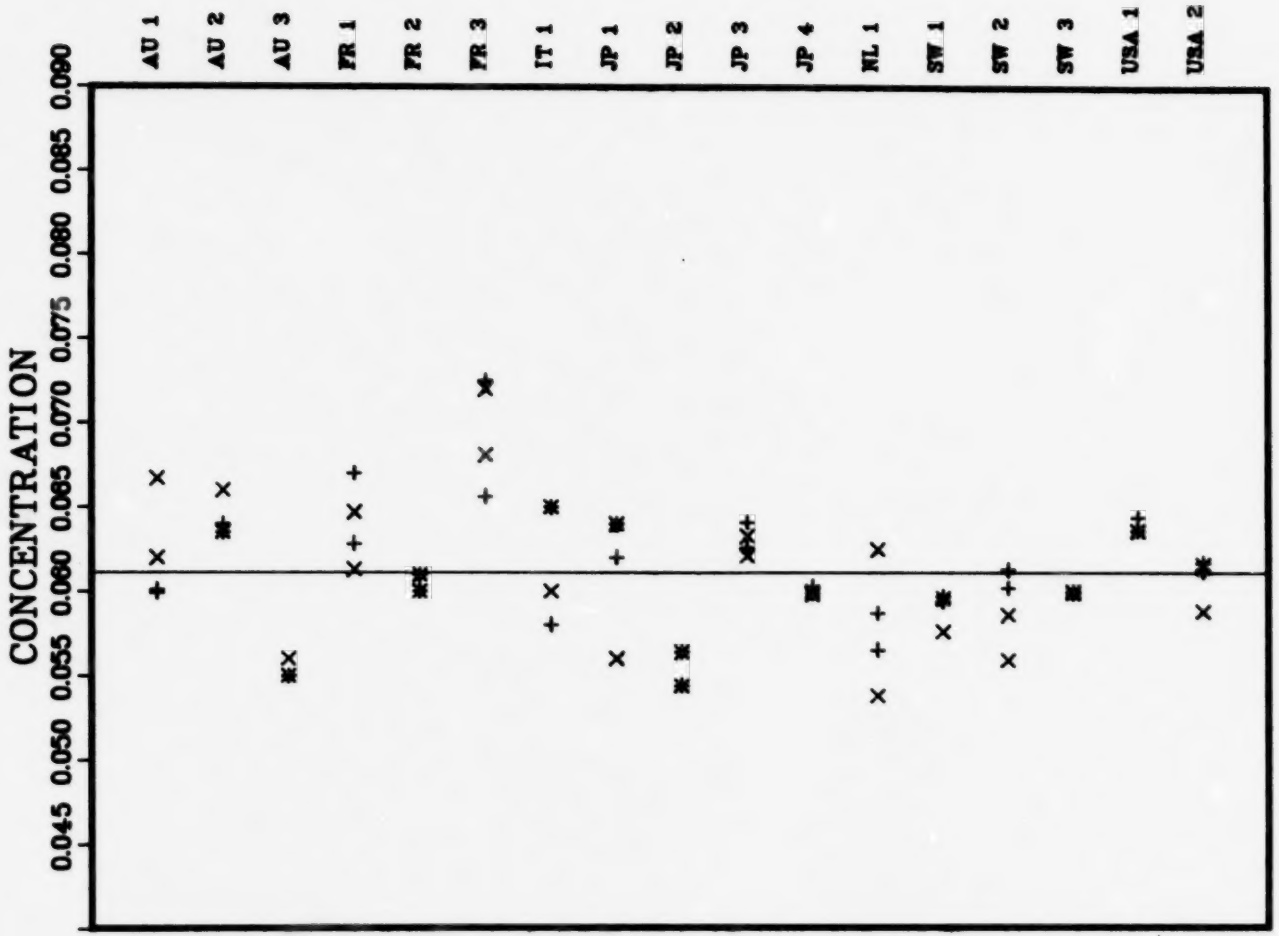

Figure 2.2.4 **IRON ORES – VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A–9 SAMPLE 81–5**

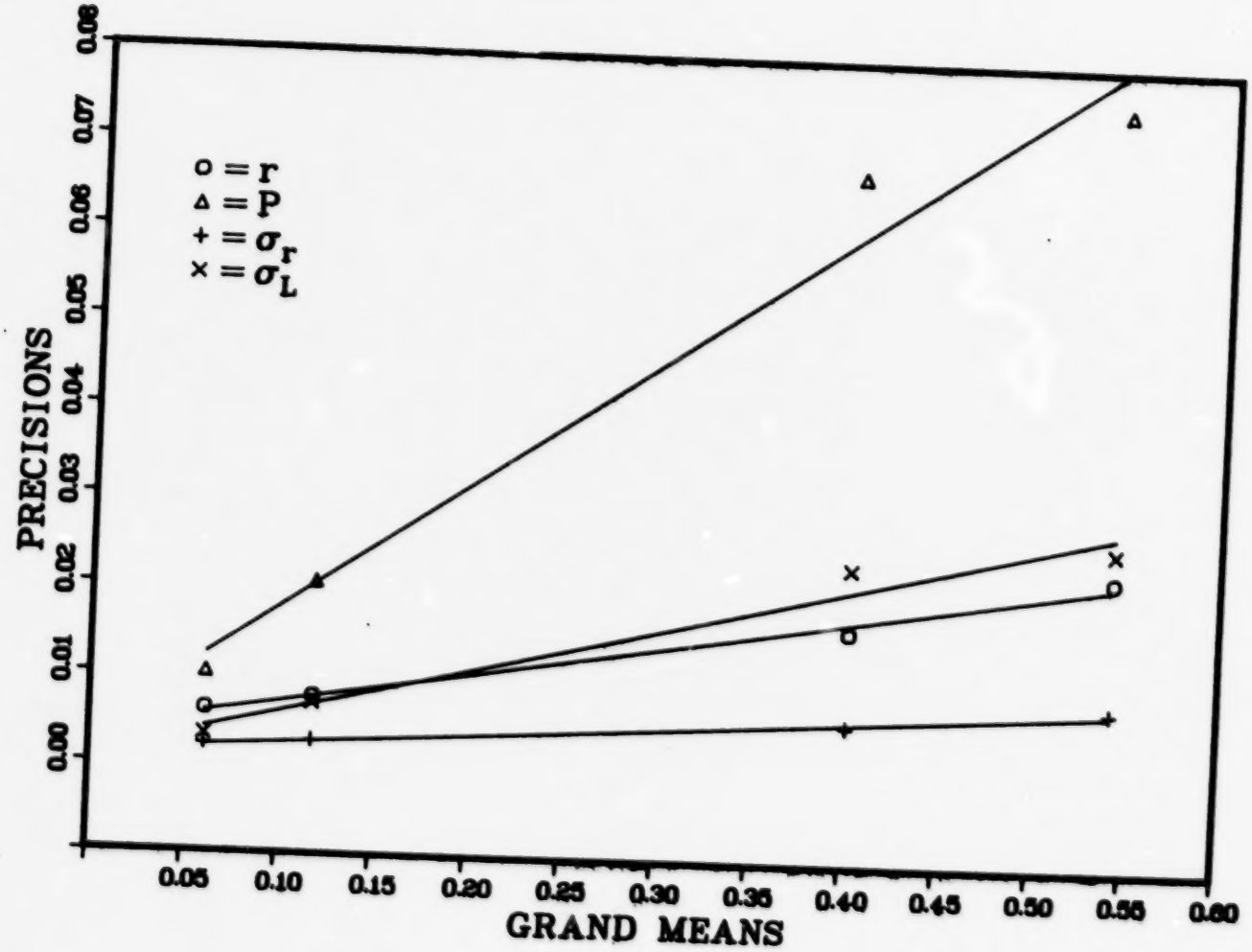

Figure 3.1 IRON ORES – VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-2

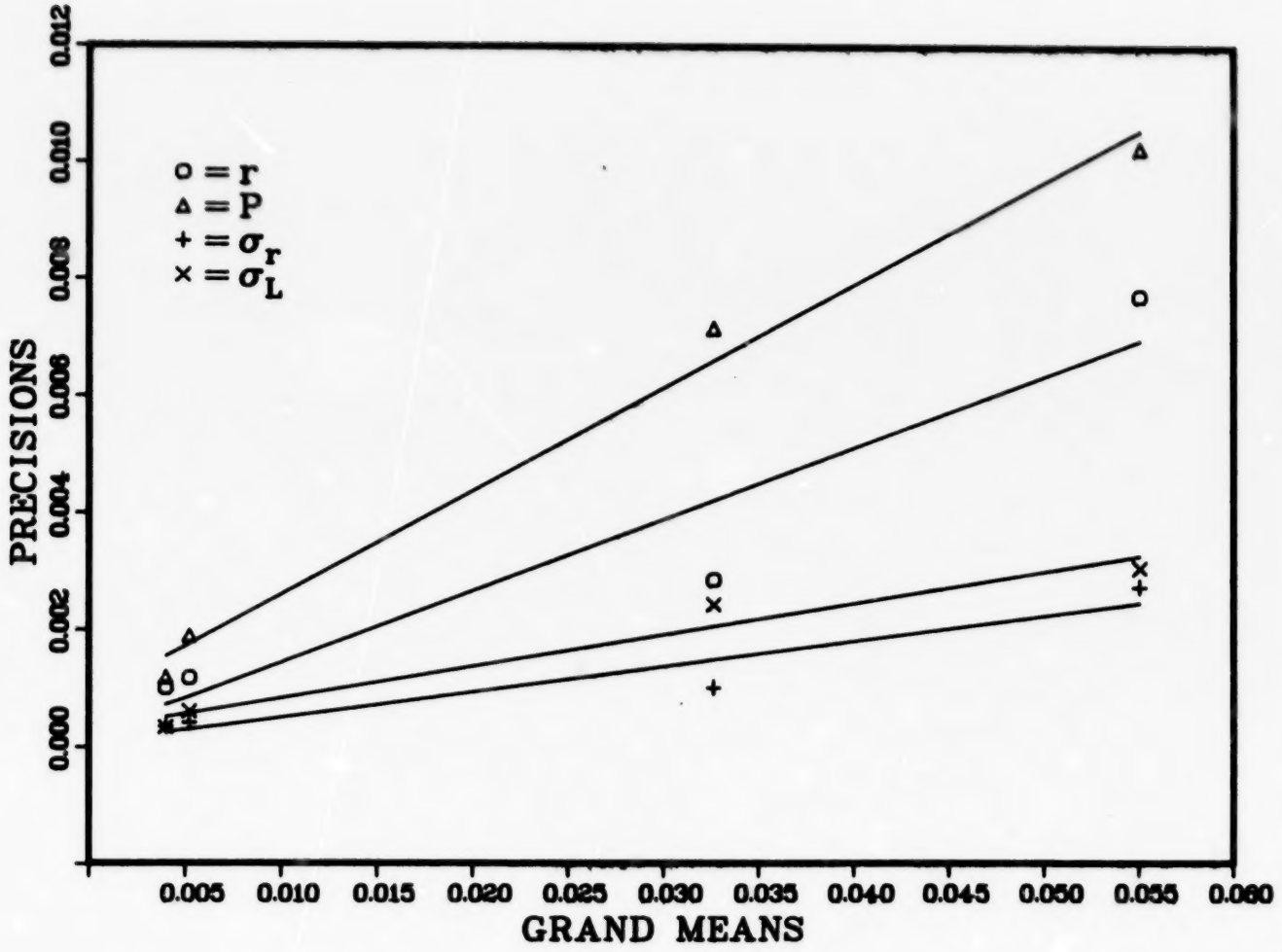

Figure 3.2 IRON ORES – VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9

APPENDIX A

ABBREVIATIONS USED IN THIS REPORT

σ_r = SIGMA-R - within laboratory standard deviation

σ_L = SIGMA-L - between laboratories standard deviation

r = R - repeatability
= 2.83 * σ_r

P = permissible tolerance between laboratories

= 2.83 $\sigma_L^2 + \sigma_r^2/2$

COUNTRY ABBREVIATIONS

AU = Australia

FR = France

IT = Italy

JP = Japan

NL = Netherlands

SW = Sweden

USA = United States

APPENDIX B

DETAILED COMPUTATION OF THE ANALYTICAL DATA

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-5

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	.0581	.0609	.05868	.2296E-05
		2	.0575	.0582		
2	AU 2	1	.0601	.0566	.05863	.1031E-04
		2	.0624	.0554		
3	AU 3	1	.0498	.0477	.04605	.1066E-04
		2	.0428	.0439		
4	FR 1	1	.0530	.0571	.05580	.4593E-05
		2	.0553	.0578		
5	FR 2	1	.0515	.0440	.05040	.7334E-04
		2	.0621	.0440		
6	FR 3	1	.0584	.0497	.05418	.2554E-04
		2	.0587	.0499		
7	JP 1	1	.0250	.0188	.02503	.3037E-03
		2	.0488	.0075		
8	JP 2	1	.0522	.0511	.05150	.3667E-06
		2	.0518	.0509		
9	JP 3	1	.0600	.0597	.05975	.7000E-07
		2	.0599	.0594		
10	JP 4	1	.0614	.0576	.06020	.3067E-05
		2	.0608	.0610		
11	NL 1	1	.0539	.0548	.05300	.5367E-05
		2	.0496	.0537		
12	SW 1	1	.0486	.0518	.05153	.4089E-05
		2	.0526	.0531		
13	SW 2	1	.0410	.0442	.05195	.1224E-03
		2	.0588	.0638		
14	SW 3	1	.0544	.0478	.05403	.1880E-04
		2	.0565	.0574		
15	USA 1	1	.0593	.0619	.06040	.5020E-05
		2	.0578	.0626		
16	USA 2	1	.0572	.0558	.05493	.5036E-05
		2	.0548	.0519		

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-5

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	59.697
CHI-SQUARE(.95,31)	44.991

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR BOTTLE VARIANCE

LAB- 7	BOTTLE- 2	C-CALC = .670	C-CRIT(2,32) = .280
LAB- 5	BOTTLE- 2	C-CALC = .391	C-CRIT(2,31) = .286

DOORNBOS' TEST FOR BOTTLE MEANS

LAB- 7	BOTTLE- 1	T-CALC = 4.588	T-CRIT(.00156,30) = 3.479
LAB- 7	BOTTLE- 2	T-CALC = 5.221	T-CRIT(.00161,29) = 3.479

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 5	COCHRAN'S TEST FOR BOTTLE VARIANCE
LAB- 7	DOORNBOS' TEST FOR BOTTLE MEANS

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-5

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	13	.90156214E-03	.69350934E-04
BETWEEN BOTTLES	14	.44489500E-03	.31778214E-04
WITHIN BOTTLES	28	.20806000E-03	.74307143E-05
TOTAL	55	.15545171E-02	

F-CALC = 4.277 F-CRIT(.95;14,28) = 2.064

BETWEEN-BOTTLE VARIANCE IS SIGNIFICANT
TEST SAMPLE IS PRESUMED TO BE INHOMOGENEOUS

ONE-WAY ANOVA CAN NOT BE PERFORMED
PRECISION ESTIMATES FROM TWO-WAY ANOVA

REPEATABILITY VARIANCE	.74307143E-05
REPEATABILITY STD. DEV.	.00272593
BETWEEN-LABORATORIES VARIANCE	.93931799E-05
BETWEEN-LABORATORIES STD. DEV.	.00306483
REPEATABILITY	.00771439
PERMISSIBLE TOLERANCE BETWEEN LABS	.01024622
GRAND MEAN	.05504286
VARIANCE OF THE GRAND MEAN	.12384095E-05

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 79-14

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	.0356	.0358	.03543	.4825E-06
		2	.0359	.0344		
2	AU 2	1	.0355	.0333	.03435	.1050E-05
		2	.0349	.0337		
3	AU 3	1	.0275	.0281	.03003	.6676E-05
		2	.0324	.0321		
4	FR 1	1	.0334	.0334	.03280	.1607E-05
		2	.0309	.0335		
5	FR 2	1	.0251	.0238	.02023	.2675E-04
		2	.0140	.0180		
6	FR 3	1	.0300	.0301	.02990	.1667E-06
		2	.0293	.0302		
7	JP 1	1	.0186	.0138	.01400	.1629E-04
		2	.0088	.0148		
8	JP 2	1	.0265	.0266	.02723	.7158E-06
		2	.0283	.0275		
9	JP 3	1	.0364	.0342	.03575	.1103E-05
		2	.0364	.0360		
10	JP 4	1	.0338	.0353	.03495	.1470E-05
		2	.0365	.0342		
11	NL 1	1	.0299	.0312	.02943	.4129E-05
		2	.0265	.0301		
12	SW 1	1	.0327	.0338	.03355	.8033E-06
		2	.0347	.0330		
13	SW 2	1	.0300	.0304	.03525	.3484E-04
		2	.0392	.0414		
14	SW 3	1	.0335	.0335	.03288	.5625E-06
		2	.0325	.0320		
15	USA 1	1	.0344	.0343	.03495	.5633E-06
		2	.0359	.0352		
16	USA 2	1	.0311	.0300	.03103	.5092E-06
		2	.0316	.0314		

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 79-14

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	35.486
CHI-SQUARE(.95,27)	40.119

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DOORNBOS' TEST FOR BOTTLE MEANS

LAB- 7	BOTTLE- 2	T-CALC = 3.677	T-CRIT(.00156,30) = 3.479
LAB- 5	BOTTLE- 2	T-CALC = 3.495	T-CRIT(.00161,29) = 3.479
LAB- 7	BOTTLE- 1	T-CALC = 4.680	T-CRIT(.00167,28) = 3.479

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 4	ZERO WITHIN-BOTTLE VARIANCE
LAB- 5	DOORNBOS' TEST FOR BOTTLE MEANS
LAB- 7	DOORNBOS' TEST FOR BOTTLE MEANS
LAB-14	ZERO WITHIN-BOTTLE VARIANCE

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 79-14

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	11	.38110229E-03	.34645663E-04
BETWEEN BOTTLES	12	.13325250E-03	.11104375E-04
WITHIN BOTTLES	24	.24265000E-04	.10110417E-05
TOTAL	47	.53861979E-03	

F-CALC =10.983 F-CRIT(.95;12,24) = 2.183

BETWEEN-BOTTLE VARIANCE IS SIGNIFICANT
TEST SAMPLE IS PRESUMED TO BE INHOMOGENEOUS

ONE-WAY ANOVA CAN NOT BE PERFORMED
PRECISION ESTIMATES FROM TWO-WAY ANOVA

REPEATABILITY VARIANCE	.10110417E-05
REPEATABILITY STD. DEV.	.00100551
BETWEEN-LABORATORIES VARIANCE	.58853220E-05
BETWEEN-LABORATORIES STD. DEV.	.00242597
REPEATABILITY	.00284558
PERMISSIBLE TOLERANCE BETWEEN LABS	.00715427
GRAND MEAN	.03265208
VARIANCE OF THE GRAND MEAN	.72178464E-06

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 79-16

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	.0055	.0050	.00513	.2292E-06
		2	.0045	.0055		
2	AU 2	1	.0063	.0052	.00570	.4867E-06
		2	.0063	.0050		
3	AU 3	1	.0049	.0049	.00453	.2025E-06
		2	.0043	.0040		
4	FR 1	1	.0040	.0048	.00453	.2625E-06
		2	.0042	.0051		
5	FR 2	1	.0048	.0055	.00485	.2433E-06
		2	.0043	.0048		
6	FR 3	1	.0051	.0047	.00490	.1333E-06
		2	.0053	.0045		
7	JP 1	1	.0030	.0023	.00195	.7767E-06
		2	.0015	.0010		
8	JP 2	1	.0052	.0051	.00483	.1692E-06
		2	.0043	.0047		
9	JP 3	1	.0047	.0048	.00483	.9167E-08
		2	.0049	.0049		
10	JP 4	1	.0057	.0058	.00545	.1233E-06
		2	.0052	.0051		
11	NL 1	1	.0055	.0055	.00515	.1700E-06
		2	.0047	.0049		
12	SW 1	1	.0064	.0062	.00645	.3667E-07
		2	.0066	.0066		
13	SW 2	1	.0072	.0058	.00670	.4400E-06
		2	.0072	.0066		
14	SW 3	1	.0050	.0050	.00500	0.
		2	.0050	.0050		
15	USA 1	1	.0054	.0054	.00540	0.
		2	.0054	.0054		
16	USA 2	1	.0050	.0053	.00498	.5583E-07
		2	.0048	.0048		

IRON ORES - VANADIUM BY AAS METH[illegible] 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 79-16

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	10.291
CHI-SQUARE(.95,17)	27.593

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DOORNBOS' TEST FOR BOTTLE MEANS

LAB- 7	BOTTLE- 2	T-CALC = 4.802	T-CRIT(.00156,30) = 3.479
LAB- 7	BOTTLE- 1	T-CALC = 3.834	T-CRIT(.00161,29) = 3.479

LIST OF LAB RATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 3	ZERO WITHIN-BOTTLE VARIANCE
LAB- 7	DOORNBOS' TEST FOR BOTTLE MEANS
LAB- 9	ZERO WITHIN-BOTTLE VARIANCE
LAB-11	ZERO WITHIN-BOTTLE VARIANCE
LAB-12	ZERO WITHIN-BOTTLE VARIANCE
LAB-14	ZERO WITHIN-BOTTLE VARIANCE
LAB-15	ZERO WITHIN-BOTTLE VARIANCE
LAB-16	ZERO WITHIN-BOTTLE VARIANCE

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 79-16

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	7	.13394688E-04	.19135268E-05
BETWEEN BOTTLES	8	.14375000E-05	.17968750E-06
WITHIN BOTTLES	16	.48250000E-05	.30156250E-06
TOTAL	31	.19657188E-04	

F-CALC = .596 F-CRIT(.95; 8,16) = 2.591

TEST SAMPLE IS HOMOGENEOUS

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 79-16

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	16.313
CHI-SQUARE(.95,13)	22.367

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DIXON'S TEST FOR LABORATORY MEANS

LAB- 7 R = .687 D-CRIT(16) = .546

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 7 DIXON'S TEST FOR LABORATORY MEANS

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 79-16

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	14	.22632333E-04	.16165952E-05
WITHIN LABS	45	.76850000E-05	.17077778E-06
TOTAL	59	.30317333E-04	

REPEATABILITY VARIANCE	.17077778E-06
REPEATABILITY STD. DEV.	.00041325
BETWEEN-LABORATORIES VARIANCE	.36145437E-06
BETWEEN-LABORATORIES STD. DEV.	.00060121
REPEATABILITY	.00116951
PERMISSIBLE TOLERANCE BETWEEN LABS	.00189175
GRAND MEAN	.00522667
VARIANCE OF THE GRAND MEAN	.26943254E-07

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 79-17

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	.0040	.0042	.00405	.3667E-07
		2	.0038	.0042		
2	AU 2	1	.0054	.0042	.00463	.3225E-06
		2	.0047	.0042		
3	AU 3	1	.0034	.0039	.00405	.2567E-06
		2	.0045	.0044		
4	FR 1	1	.0030	.0039	.00325	.3233E-06
		2	.0035	.0026		
5	FR 2	1	.0040	.0035	.00370	.1267E-06
		2	.0040	.0033		
6	FR 3	1	.0041	.0037	.00390	.1333E-06
		2	.0043	.0035		
7	JP 1	1	.0010	.0015	.00133	.5583E-07
		2	.0013	.0015		
8	JP 2	1	.0036	.0036	.00363	.2500E-08
		2	.0037	.0036		
9	JP 3	1	.0035	.0037	.00363	.9167E-08
		2	.0036	.0037		
10	JP 4	1	.0046	.0038	.00438	.1625E-06
		2	.0047	.0044		
11	NL 1	1	.0046	.0041	.00418	.1092E-06
		2	.0038	.0042		
12	SW 1	1	.0042	.0044	.00438	.5583E-07
		2	.0047	.0042		
13	SW 2	1	.0046	.0028	.00400	.1200E-05
		2	.0052	.0034		
14	SW 3	1	.0042	.0037	.00403	.4917E-07
		2	.0041	.0041		
15	USA 1	1	.0042		.00423	.3333E-08
		2	.0043	.0042		
16	USA 2	1	.0036	.0037	.00440	.7667E-06
		2	.0050	.0053		

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 79-17

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	20.726
CHI-SQUARE(.95,25)	37.660

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DOORNBOS' TEST FOR BOTTLE MEANS

LAB- 7	BOTTLE- 1	T-CALC = 3.992	T-CRIT(.00167,28) = 3.479
LAB- 7	BOTTLE- 2	T-CALC = 5.666	T-CRIT(.00172,27) = 3.479

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 7	DOORNBOS' TEST FOR BOTTLE MEANS
LAB- 8	ZERO WITHIN-BOTTLE VARIANCE
LAB-14	ZERO WITHIN-BOTTLE VARIANCE
LAB-15	NUMBER OF BOTTLES OR REPLICATES INCORRECT

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 79-17

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	11	.65906250E-05	.59914773E-06
BETWEEN BOTTLES	12	.38225000E-05	.31854167E-06
WITHIN BOTTLES	24	.66850000E-05	.27854167E-06
TOTAL	47	.17098125E-04	

F-CALC = 1.144 F-CRIT(.95;12,24) = 2.183

TEST SAMPLE IS HOMOGENEOUS

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 79-17

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	41.202
CHI-SQUARE(.95,15)	25.000

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB-13	C-CALC =	.332	C-CRIT(4,16) =	.262
LAB-16	C-CALC =	.318	C-CRIT(4,15) =	.275

DIXON'S TEST FOR LABORATORY MEANS

LAB- 7	R =	.754	D-CRIT(16) =	.546

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 7	DIXON'S TEST FOR LABORATORY MEANS
LAB-13	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-16	COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

IRON ORES - VANADIUM BY AAS METHOD 1(WITH EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 79-17

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	12	.67700490E-05	.56417075E-06
WITHIN LABS	38	.47691667E-05	.12550439E-06
TOTAL	50	.11539216E-04	

REPEATABILITY VARIANCE	.12550439E-06
REPEATABILITY STD. DEV.	.00035427
BETWEEN-LABORATORIES VARIANCE	.11185992E-06
BETWEEN-LABORATORIES STD. DEV.	.00033445
REPEATABILITY	.00100257
PERMISSIBLE TOLERANCE BETWEEN LABS	.00118256
GRAND MEAN	.00399608
VARIANCE OF THE GRAND MEAN	.11105178E-07

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 76-17

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	.5601	.5474	.55303	.7025E-04
		2	.5443	.5603		
2	AU 2	1	.5660	.5670	.56800	.2333E-04
		2	.5750	.5640		
3	AU 3	1	.5170	.5080	.50900	.4333E-04
		2	.5010	.5100		
4	FR 1	1	.5555	.5642	.56135	.2103E-04
		2	.5600	.5657		
5	FR 2	1	.5450	.5410	.54450	.5667E-05
		2	.5460	.5460		
6	FR 3	1	.5990	.6254	.61200	.1644E-03
		2	.6033	.6203		
7	IT 1	1	.5500	.5260	.53100	.1720E-03
		2	.5280	.5200		
8	JP 1	1	.5550	.5520	.55325	.2250E-05
		2	.5520	.5540		
9	JP 2	1	.4948	.5071	.50283	.2991E-04
		2	.5046	.5048		
10	JP 3	1	.5465	.5394	.54105	.1426E-04
		2	.5379	.5404		
11	JP 4	1	.5250	.5383	.53155	.5723E-04
		2	.5250	.5379		
12	NL 1	1	.5121	.5441	.53240	.2218E-03
		2	.5304	.5430		
13	SW 1	1	.5085	.5081	.50635	.5523E-05
		2	.5036	.5052		
14	SW 2	1	.5377	.5479	.54215	.2031E-04
		2	.5396	.5434		
15	SW 3	1	.5359	.5391	.53640	.3647E-05
		2	.5360	.5346		
16	USA 1	1	.5640	.5697	.56623	.7242E-05
		2	.5642	.5670		
17	USA 2	1	.5528	.5393	.53505	.2393E-03
		2	.5156	.5325		

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 76-17

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	34.608
CHI-SQUARE(.95,31)	44.991

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DOORNBOS' TEST FOR BOTTLE MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 5 ZERO WITHIN-BOTTLE VARIANCE

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 76-17

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	15	.44308597E-01	.29539065E-02
BETWEEN BOTTLES	16	.87531750E-03	.54707344E-04
WITHIN BOTTLES	32	.24120350E-02	.75376094E-04
TOTAL	63	.47595950E-01	

F-CALC = .726 F-CRIT(.95;16,32) = 1.972

TEST SAMPLE IS HOMOGENEOUS

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 76-17

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	40.054
CHI-SQUARE(.95,16)	26.301

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

NONE REJECTED

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

ALL RESULTS WERE USED EXCEPT INDIVIDUAL REPLICATES LISTED BELOW

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 76-17

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	16	.44322166E-01	.27701353E-02
WITHIN LABS	51	.33043525E-02	.64791225E-04
TOTAL	67	.47626518E-01	

REPEATABILITY VARIANCE	.64791225E-04
REPEATABILITY STD. DEV.	.00804930
BETWEEN-LABORATORIES VARIANCE	.67633603E-03
BETWEEN-LABORATORIES STD. DEV.	.02600646
REPEATABILITY	.02277952
PERMISSIBLE TOLERANCE BETWEEN LABS	.07534030
GRAND MEAN	.54271324
VARIANCE OF THE GRAND MEAN	.40737285E-04

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-3

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	.4061	.4080	.41055	.4298E-04
		2	.4078	.4203		
2	AU 2	1	.4210	.4180	.41950	.9667E-05
		2	.4230	.4160		
3	AU 3	1	.3540	.3500	.34750	.3567E-04
		2	.3400	.3460		
4	FR 1	1	.4202	.4301	.42258	.3638E-04
		2	.4159	.4241		
5	FR 2	1	.4110	.4100	.40950	.9667E-05
		2	.4050	.4120		
6	FR 3	1	.4042	.4511	.43540	.1089E-02
		2	.4121	.4742		
7	IT 1	1	.4100	.3920	.39825	.2042E-03
		2	.4100	.3810		
8	JP 1	1	.4340	.4340	.43550	.3667E-05
		2	.4380	.4360		
9	JP 2	1	.3556	.3730	.36468	.1102E-03
		2	.3556	.3745		
10	JP 3	1	.4291	.4311	.42783	.8783E-05
		2	.4269	.4242		
11	JP 4	1	.3985	.3903	.39425	.2138E-04
		2	.3980	.3902		
12	NL 1	1	.3863	.4002	.39413	.1183E-03
		2	.3837	.4063		
13	SW 1	1	.4047	.4040	.40318	.2163E-05
		2	.4026	.4014		
14	SW 2	1	.3876	.3986	.39538	.4687E-04
		2	.3922	.4031		
15	SW 3	1	.3979	.3976	.39565	.5910E-05
		2	.3934	.3937		
16	USA 1	1	.4029	.4098	.40705	.1196E-04
		2	.4100	.4055		
17	USA 2	1	.3433	.3660	.37048	.4573E-03
		2	.3788	.3938		

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-3

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	49.854
CHI-SQUARE(.95,31)	44.991

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR BOTTLE VARIANCE

LAB- 6	BOTTLE- 2	C-CALC =	.376	C-CRIT(2,32) =	.280
LAB- 6	BOTTLE- 1	C-CALC =	.344	C-CRIT(2,31) =	.286

DOORNBOS' TEST FOR BOTTLE MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 6	COCHRAN'S TEST FOR BOTTLE VARIANCE
LAB- 8	ZERO WITHIN-BOTTLE VARIANCE

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-3

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	14	.27232334E-01	.19451667E-02
BETWEEN BOTTLES	15	.12627375E-02	.84182500E-04
WITHIN BOTTLES	30	.21017450E-02	.70058167E-04
TOTAL	59	.30596817E-01	

F-CALC = 1.202 F-CRIT(.95;15,30) = 2.015

TEST SAMPLE IS HOMOGENEOUS

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-3

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	64.399
CHI-SQUARE(.95,16)	26.301

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB- 6	C-CALC =	.492	C-CRIT(4,17) =	.250
LAB-17	C-CALC =	.406	C-CRIT(4,16) =	.262
LAB- 7	C-CALC =	.306	C-CRIT(4,15) =	.275

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 6	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB- 7	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-17	COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-3

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	13	.28978614E-01	.22291242E-02
WITHIN LABS	42	.13908650E-02	.33115833E-04
TOTAL	55	.30369479E-01	

REPEATABILITY VARIANCE	.33115833E-04
REPEATABILITY STD. DEV.	.00575464
BETWEEN-LABORATORIES VARIANCE	.54900209E-03
BETWEEN-LABORATORIES STD. DEV.	.02343079
REPEATABILITY	.01628562
PERMISSIBLE TOLERANCE BETWEEN LABS	.06730166
GRAND MEAN	.40194643
VARIANCE OF THE GRAND MEAN	.39805789E-04

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-4

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	.1201	.1271	.12128	.1611E-04
		2	.1179	.1200		
2	AU 2	1	.1270	.1210	.12425	.6250E-05
		2	.1250	.1240		
3	AU 3	1	.1070	.1000	.10850	.4300E-04
		2	.1150	.1120		
4	FR 1	1	.1255	.1288	.12640	.2580E-05
		2	.1255	.1258		
5	FR 2	1	.1180	.1200	.11900	.1333E-05
		2	.1180	.1200		
6	FR 3	1	.1229	.1398	.13308	.7557E-04
		2	.1288	.1408		
7	IT 1	1	.1080	.1060	.10475	.1158E-04
		2	.1000	.1050		
8	JP 1	1	.1260	.1230	.12425	.1583E-05
		2	.1240	.1240		
9	JP 2	1	.1091	.1109	.11023	.6225E-06
		2	.1106	.1103		
10	JP 3	1	.1217	.1222	.12155	.1657E-05
		2	.1226	.1197		
11	JP 4	1	.1175	.1150	.11603	.1649E-05
		2	.1167	.1149		
12	NL 1	1	.1076	.1168	.11263	.2423E-04
		2	.1092	.1169		
13	SW 1	1	.1012	.1015	.10425	.1591E-04
		2	.1098	.1045		
14	SW 2	1	.1157	.1217	.11958	.1010E-04
		2	.1183	.1226		
15	SW 3	1	.1159	.1159	.11593	.2500E-08
		2	.1160	.1159		
16	USA 1	1	.1219	.1229	.12170	.1947E-05
		2	.1223	.1197		
17	USA 2	1	.1266	.1216	.12553	.1625E-04
		2	.1307	.1232		

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-4

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	32.289
CHI-SQUARE(.95,29)	42.565

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DOORNBOS' TEST FOR BOTTLE MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 8	ZERO WITHIN-BOTTLE VARIANCE
LAB-15	ZERO WITHIN-BOTTLE VARIANCE

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-4

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	14	.39280440E-02	.28057457E-03
BETWEEN BOTTLES	15	.20482750E-03	.13655167E-04
WITHIN BOTTLES	30	.48154500E-03	.16051500E-04
TOTAL	59	.46144165E-02	

F-CALC = .851 F-CRIT(.95;15,30) = 2.015

TEST SAMPLE IS HOMOGENEOUS

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-4

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	56.923
CHI-SQUARE(.95,16)	26.301

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB- 6	C-CALC =	.328	C-CRIT(4,17) =	.250
LAB- 3	C-CALC =	.278	C-CRIT(4,16) =	.262

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 3	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB- 6	COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-4

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	14	.28300193E-02	.20214424E-03
WITHIN LABS	45	.33542250E-03	.74538333E-05
TOTAL	59	.31654418E-02	

REPEATABILITY VARIANCE	.74538333E-05
REPEATABILITY STD. DEV.	.00273017
BETWEEN-LABORATORIES VARIANCE	.48672601E-04
BETWEEN-LABORATORIES STD. DEV.	.00697658
REPEATABILITY	.00772638
PERMISSIBLE TOLERANCE BETWEEN LABS	.02048567
GRAND MEAN	.11782167
VARIANCE OF THE GRAND MEAN	.33690706E-05

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-5

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	.0620	.0667	.06218	.9996E-05
		2	.0599	.0601		
2	AU 2	1	.0660	.0635	.06425	.1417E-05
		2	.0640	.0635		
3	AU 3	1	.0560	.0550	.05525	.2500E-06
		2	.0550	.0550		
4	FR 1	1	.0613	.0647	.06395	.6070E-05
		2	.0628	.0670		
5	FR 2	1	.0600	.0610	.06050	.3333E-06
		2	.0600	.0610		
6	FR 3	1	.0681	.0720	.06955	.1080E-04
		2	.0656	.0725		
7	IT 1	1	.0650	.0600	.06200	.1267E-04
		2	.0650	.0580		
8	JP 1	1	.0560	.0640	.06150	.1433E-04
		2	.0620	.0640		
9	JP 2	1	.0544	.0564	.05540	.1333E-05
		2	.0544	.0564		
10	JP 3	1	.0633	.0621	.06303	.7558E-06
		2	.0641	.0626		
11	JP 4	1	.0598	.0600	.06000	.4667E-07
		2	.0599	.0603		
12	NL 1	1	.0538	.0625	.05788	.1352E-04
		2	.0565	.0587		
13	SW 1	1	.0576	.0596	.05908	.9825E-06
		2	.0597	.0594		
14	SW 2	1	.0559	.0586	.05900	.5500E-05
		2	.0602	.0613		
15	SW 3	1	.0600	.0599	.05995	.3333E-08
		2	.0600	.0599		
16	USA 1	1	.0636	.0636	.06380	.1600E-06
		2	.0644	.0636		
17	USA 2	1	.0616	.0588	.06083	.1869E-05
		2	.0612	.0617		

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-5

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	39.937
CHI-SQUARE(.95,29)	42.565

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DOORNBOS' TEST FOR BOTTLE MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 3	ZERO WITHIN-BOTTLE VARIANCE
LAB-16	ZERO WITHIN-BOTTLE VARIANCE

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-5

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	14	.59682433E-03	.42630310E-04
BETWEEN BOTTLES	15	.50012500E-04	.33341667E-05
WITHIN BOTTLES	30	.18888500E-03	.62961667E-05
TOTAL	59	.83572183E-03	

F-CALC = .530 F-CRIT(.95;15,30) = 2.015

TEST SAMPLE IS HOMOGENEOUS _

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-5

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	60.301
CHI-SQUARE(.95,16)	26.301

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

NONE REJECTED

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

ALL RESULTS WERE USED EXCEPT INDIVIDUAL REPLICATES LISTED BELOW

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

IRON ORES - VANADIUM BY AAS METHOD 2(WITHOUT EXTRACTION) ISO/TC102/SC2/WG 18A-9
SAMPLE 81-5

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	16	.76456471E-03	.47785294E-04
WITHIN LABS	51	.24012750E-03	.47083824E-05
TOTAL	67	.10046922E-02	

REPEATABILITY VARIANCE	.47083824E-05
REPEATABILITY STD. DEV.	.00216988
BETWEEN-LABORATORIES VARIANCE	.10769228E-04
BETWEEN-LABORATORIES STD. DEV.	.00328165
REPEATABILITY	.00614076
PERMISSIBLE TOLERANCE BETWEEN LABS	.01025203
GRAND MEAN	.06106618
VARIANCE OF THE GRAND MEAN	.70272491E-06

POOR

QUALITY

ORIGINAL